PATENT SPECIFICATION

920,261

DRAWINGS ATTACHED.

Date of Application and filing Complete Specification :
Sept. 1, 1959. *No. 29763/59.*

Application made in United States of America (No. 758,381)
on Sept. 2, 1958.

Complete Specification Published : March 6, 1963.

Index at Acceptance :—Classes 49, B1(B:L), E1 ; 1(2), AC3(A11:B11:C11), AD37, AG50D37 ; and 87(2), A1L.

International Classification :—A23b, l. B29d. C01c, d.

COMPLETE SPECIFICATION.

Process and Apparatus for Agglomerating Pulverulent Materials and Products thereof.

ERRATA

SPECIFICATION NO. 920,261

Page 2, line 12, *for* "or" *read* "of"

Page 3, line 17, *for* "with in" *read* "within"

Page 3, line 101, *after* "mixtures" *insert* ")"

Page 5, line 88, *for* "sectond" *read* "second"

Page 5, line 114, *for* "18" *read* "19"

Page 6, line 18, *for* "9½" *read* "19½"

Page 6, *after* line 40, *insert* "Temperatures of vapor-gas fed"

THE PATENT OFFICE,
16th April 1963

DS 72955/1(13)/R.109 200 4/63 PL

carrying out the above process.

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Process and Apparatus for Agglomerating Pulverulent Materials and Products thereof.

We, THE PILLSBURY COMPANY, a Corporation organized under the laws of the State of Delaware, United States of America, of Minneapolis, Minnesota, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the art of agglomerating pulverulent materials and more particularly to both a process and an apparatus for capacity production of readily soluble or dispersible agglomerates.

The present invention provides a process of agglomerating pulverulent material including the steps of continuously dispersing said material so as to fluidize the particles of said material and form a fluidized bed of said material in which the particles are dispersed and suspended in intimate relationship with one another and make repeated contact with one another, maintaining said material within the confines of said bed while moving the bed over a supporting medium, and subjecting the fluidized particles of at least a part of said moving bed to a condition changing agent so as to form adhesive agglomerating surfaces on the particles and thereby effect agglomeration thereof.

The invention also provides apparatus for carrying out the above process.

Agglomeration broadly defined consists in the formation of clusters of very small individual particles by producing adhesive films on the surfaces of the individual particles and then uniting multiplicities of the same to form a lacy network. The lacy network thereby produced defines voids or interstices between the original particles thereby causing very rapid permeation and absorption of water or other liquids and consequently rendering the product readily soluble or dispersible.

Regardless of the end objectives, the behaviour of the particular powdered material during agglomeration procedure varies substantially. As a consequence the type of equipment and the process steps employed for agglomerating the numerous types of pulverulent material becomes important in relation to the adhesive, the melting point and other qualities of the particular material.

In agglomerating a number of powdered materials, addition of small quantities of adhesive substances is often useful. However, many pulverulent materials, including a number of food-ingredients used in packaged mixes and including many powdered chemicals, inherently develop adequate surface adhesiveness for the production of agglomerates thereof when subjected to moisture or surface condensation of vapors. Surface contact of the adhesive particles and subsequent hardening of the adhesive substances are of course requisite in the formation of agglomerates.

The prior art processes and apparatus have limitations and disadvantages in the production of agglomerates from many of the available pulverulent materials and particularly in the case of materials possessing inherent adhesive characteristics. In general, the prior art methods and apparatus for agglomerating powdered material fall into two classes, to wit:—

(1) Methods and apparatus which subject the powdered material during rapid mechanical agitation thereof to steam or very humid air. Revolving beater arms or equivalent mechanical agitators are generally employed with means for distributing the moistening medium on all of the solid particles in agitated and dispersed state; and

(2) Methods and apparatus which atomize the pulverulent material into an enveloping atmosphere of steam or humid air.

In the operation of both classes of said prior art, the total quantity of solids introduced and initially formed into agglomerates becomes sticky and the particles come into contact with walls, passages, moving parts (class 1) and other components of the apparatus before the adhesive substances are dried, thereby fouling the apparatus and producing undesirable incrustations on parts thereof. Such fouling is greatly accentuated where the powdered material is inherently sticky.

In actual practice of prior art methods and apparatus it is found that the space requirement for production of a commercially desirable capacity of agglomerates is extremely large in comparison with that required for carrying out our improved process. The prior art methods are further objectionable because of unduly high power requirements and comparatively low thermal efficiencies.

We are concerned therefore with the production at high capacity of agglomerates from numerous pulverulent materials carried out in minimum space requirements and with the elimination of fouling and the forming of incrustations on the walls and working parts of the apparatus.

It is a general object to provide a relatively inexpensive and highly efficient process and apparatus for agglomerating pulverulent materials of manifold characteristics and compositions which will eliminate the said shortcomings of the prior art processes and apparatus.

A further object is the provision of apparatus which in operation will eliminate fouling of equipment and accumulation of incrustations and which will operate at high capacity and efficiency with low power and space requirements.

These and other objects and advantages of the invention will more fully appear from the following description made in connection with the accompanying drawings wherein like reference characters refer to the same parts throughout the several views and in which an exemplary form of agglomerating apparatus, well suited to carry out our improved process commercially and at high capacity, is illustrated in the drawings, and in which:—

Figure 1 is a top plan view of an apparatus, particularly adapted for carrying out our process, certain hidden portions thereof being shown in dotted line and diagrammatically illustrated;

Figure 2 is a vertical section taken on the line 2—2 of Figure 1;

Figure 3 is a front elevation of the apparatus, certain portions thereof unnecessary to an understanding of the invention being cut away; and

Figure 4 is a diagrammatical fragmentary vertical section taken longitudinally of the flow of material illustrating important steps in our process of agglomeration in the preferred form.

With continued reference to the drawings, our apparatus for agglomerating pulverulent products has a feed mechanism indicated generally at 10 which includes a hopper 11 communicating with a screw conveyor housing 12, which in turn, contains a screw conveyor 13 adapted to feed the pulverulent material 14 to a feed spout 15, as shown in Figures 1 and 2. The screw conveyor 13 is illustrated diagrammatically and may be journaled and rotatably driven in conventional manner by means not shown.

The dry material is delivered from the feed spout 15 to a pervious sheet 16 which may be constructed of glass cloth or similar material having a fine mesh size of from 60 to 400 filaments per linear inch capable of permitting vapor and gas to pass upwardly therethrough and exert a fluidizing effect upon pulverulent material 14 which is continuously deposited thereon. The sheet 16 is substantially planar in construction so that the material will not tend to collect at one portion thereof, or to become channeled in a particular area in the course of travel of the particles along the sheet 16. Sheet 16 is disposed across a housing frame 17 which may also provide the various chambers for pressurized fluid and for exhausting fluid, as will hereinafter more fully be explained.

As the pulverulent material leaves the sheet 16, it continues upon another sheet 18 which is also of substantially planar construction and disposed at a relative position not higher than a co-planar relationship with the first mentioned sheet. Feed material passing from the sheet 16 thus continues along the sheet 18 for treatment, as will be presently described.

As the material leaves the sheet 18, it passes to the pervious sheet 19 which is disposed at a position not higher than a co-planar relationship with the sheet 18. In the same manner, the material may pass to the next pervious sheet 20 and finally to a sheet 21 which may be of different character than the previous sheets for the purpose of

classifying the agglomerated product, retaining a portion thereof upon the surface of sheet 21 and permitting undersized agglomerates to pass through the sheet and be collected independently thereof. All of the sheets 16, 18, 19, 20 and 21 are mounted within the housing frame 17, the last being an enclosure and having the sheets co-extend for the full width thereof, as shown in Figures 1 and 2. We have found it practical to utilize a glass cloth in a continuous plane to supply the various sheet areas, as described above.

It will be noted that all of the pervious sheets are so located with respect to the top 22 and the bottom 23 of the housing frame 17 that spaced areas exist below and above the screens with in the housing frame 17. Upstanding partitions are employed to subdivide the spaces into chambers which will be more fully described below. Thus, the pervious sheet 16 is mounted at the feed end 24 of the housing frame 17 and partition 25 across the housing frame 17 defines an enclosed chamber 26 immediately below the pervious sheet 16. A corresponding upper baffle 27 extends across the housing frame 17 but terminates downwardly in an edge 28 which lies in clearance with the sheet 16 so as to permit pulverulent material to pass thereunder in the course of its treatment. A chamber 29 is created immediately above the pervious screen 16, as shown in Figure 2.

In a similar manner, an upright partition 30 is disposed across the housing frame 17 beneath the pervious sheet 18 to establish an enclosed chamber 31 thereunder. Baffle 32 is disposed across the housing frame 17 and has a downwardly terminating edge 33 which will permit material 14 to pass thereunder to the next pervious sheet 19.

Another upstanding partition 34 defines, with the housing frame 17 and the pervious sheet 19, a chamber 35 immediately below the pervious sheet 19. Baffle 36 having a downwardly terminating edge 37 defines a chamber 38 overlying pervious sheet 19.

A still further partition 39 disposed across the housing frame 17 defines a chamber 40 immediately beneath the pervious sheet 20 and a baffle 41 having a downwardly disposed edge 42 defines a chamber 43 immediately above the pervious sheet 20. At the discharge end of the housing frame 17 is end wall 44 which, with the sheet 21 and the partition 39, defines a chamber 45 and a delivery spout 46 communicates at the discharge end of the device with the same chamber 45. Immediately above the classifying sheet 21 is the chamber 47 which communicates with discharge spout 48 for delivering agglomerated material of a size which will not pass through the sheet 21.

All of the aforementioned sheets and corresponding upper and lower chambers are arranged sequentially to accommodate the pulverulent material 14 and effect a sequential treatment thereof from its introduction by feed spout 15 until its discharge from spouts 46 and 48.

The following description is concerned with certain applications of our process and apparatus where humid, heated air or other mixtures are employed as the condition-changing agent. Use of other vapors and gases for certain pulverulent materials does not in many instances require the same pretreatment of the vapor or gaseous medium nor the subsequent steps of drying and cooling.

By reference to Figure 1, it is seen that each of the chambers underlying a pervious sheet is adapted to receive pressurized air or other fluidizing gas or gas-vapor mixtures and to effect a desired treatment of that portion of the solid material which then overlies its associated pervious sheet. In the treatment of various pulverulent materials, air at room temperature may be supplied for the entire fluid treatment from a main conduit 49. Pressure may be supplied by a fan or other means (not shown). A branch conduit 50 containing a flexible joint 51 communicates with the chamber 26 and preferably supplies air at room temperature thereto. This chamber 26 and the associated pervious sheet 16 are adapted to preliminarily fluidize and/or suspend the pulverulent material 14 as it is received from the hopper mechanism 11.

A conduit 52 having a flexible joint 53 supplies air (or other fluidizing gas mixtures from the main conduit 49 to chamber 31 underlying the pervious sheet 18. This sheet with the chamber 31 provides means for the upwardly moving stream of vapor-condensing fluid medium in superheated condition to pass upwardly through the pulverulent material above sheet 18 for agglomerating the pulverulent material.

In the application and apparatus now described, a vapor line 54 (which vapor may be steam) introduces solvent vapor in predetermined proportion into the gaseous carrier medium such as air passing through conduit 52. In many instances the superheated vapor is not adequate for heating the gaseous carrier to the desired superheated temperature so ordinarily a heated element 55 is interposed between the main conduit 49 and the conduit 52 as shown in Figure 1. Heater 55 may comprise an enclosed housing 56 containing a filter 57 and conventional heating elements 58 which may be controlled by the usual means (not shown). The heated vapor-gas mixture is delivered to conduit 52 into the lower chamber 31 and permeates sheet 18 passing upwardly and uniformly into the treating chamber 33*a*.

It is important in the carrying out of our process that the temperature of the vapor-

gas mixture flowing through sheet 18 be maintained at a point considerably above the dew point of the vapor gas admixture so that the fluidized solid particles in the lower strata of the material above screen 18 will absorb the superheat and remain dry without production of condensation upon particle surfaces until contact of the impinging, upwardly flowing vapor-gas mixture is made upon surfaces of the upper strata of the flow of particles above screen 18.

The next chamber 35 of the apparatus provides a drying gas also flowing upwardly through the pervious sheet 19 into the drying chamber 38 above. The drying gas such as air may be heated and, as shown, conduit 59 which communicates through flexible joint 60 with the chamber 35 may have interposed therein a heater 61 having a housing 62, a filter 63 and a controlled heating element 64.

The next chamber 40 which underlies the pervious sheet 20 is provided as shown with a cooling gas such as air through conduit 65 and flexible coupling 66. Conduit 65 in turn, connects with the main supply conduit 49 and delivers to chamber 40 which underlies the cooling treatment chamber 43.

Since the final classifying sheet 21 (used in some applications of our process and apparatus) is not utilized for fluid treatment of the agglomerates, no fluid supply line is employed in chamber 45 which constitutes a collection chamber for the finer output product.

Since the character of the gas such as air in each of the aforementioned chambers except chamber 45 should be maintained distinct as between the pervious sheet members and the treatment chambers defined thereabove to facilitate re-use of each separate gas quantity by total or partial recirculation, we provide exhaust means to immediately take off the pressurized fluid which has been passed through the flow of fluidized material in the individual treatment chambers 29, 33*a*, 38 and 43. We accomplish this simply through separate exhaust conduits which in turn may communicate with dust collectors and fan (not shown), to wit :—

Line 68 and its flexible coupling 69 communicate through the top wall 22 of the housing with the fluidizing chamber 29. Line 70 and its flexible coupling 71 communicates with the top of chamber 33*a* to remove the gas-vapor mixture after its agglomerating action upon the material 14. Line 72 and its flexible coupling 73 provide an exhaust for chamber 38 to remove the warm vapor-gas and hot gas which have picked up solvent vapor from the agglomerated product during the drying step. Line 74 through its flexible coupling 75 communicates with chamber 43 to exhaust the gas therefrom after it has exerted a chilling and further drying influence upon the warm agglomerates. The last line 76 and its flexible coupling 77 exhaust the gas from the top of the classifying chamber 47 to remove any dust produced in the screening operation.

A preferred feature of the invention resides in the continual agitation of preferably all of the pervious sheets and chambers to keep the individual pulverulent particles as well as the agglomerates in continually dispersed and fluidized condition. The combined agitation of the pervious sheets together with the upward flow of the various pressurized gas and vapor-gas streams effects treatment of the particles and the agglomerates in a fluidized bed and contributes to maintain the entire machine in non-fouling condition. The mechanism as shown for effecting the rapid vibration comprises supporting struts 78 pivotally secured to the bottom 23 of the housing frame 17 at respective points 79 and comprises also a fixed supporting base 80 to which the lower ends of said struts are pivoted at points 81 (see Figure 2). The several struts 78 are disposed in parallel relationship and may be angled as shown so that upward swinging of the struts produces a forward movement of the entire housing frame including the pervious sheets and chamber-defining vertical partitions mounted therein. Rapid vibration is imparted through the struts 78 by suitable means such as a reciprocating arm 82 which may be pivotally secured at one end 83 to one of the arms 78 or, as shown, to the bottom 23 of the housing frame 17. The opposite end of the arm 82 is secured pivotally at a point 84 to an eccentric wheel 85 which is journaled with respect to base 80 and which is driven through a belt 86 by a motor 87.

As used herein the term "condition-changing agent" is intended to include any medium which is introduced into the bed of fluidized particles and which develops adhesive films from or on said particles on the outer surfaces thereof. The preferred operation of our process and apparatus requires for the production of adhesive films the controlled passage of the condition changing agent in the form of a superheated vapor or gaseous mixture containing a superheated vapor (hereinafter referred to as "vapor-gas"), upwardly through the permeable supporting medium or sheet 18 of the apparatus, said vapor-gas containing :—

(1) Gas, which in this Specification and the appended claims means an essentially inert gas which serves as a carrier for the agglomerating vapor and which transfers heat to the solids being treated and preferably provides the force necessary to fluidize or sufficiently agitate the bed of solids so as to cause the solid particles to be partially suspended and dispersed in the bed. The requisite gas in most cases is not condensable within the range of temperatures employed

and is inert toward the material being treated.

(2) Vapor, which is either condensable or absorbable on the surfaces of the particles being agglomerated within the range of temperatures employed and, when so condensed and/or absorbed upon said surfaces, causes adhesive films to be formed so that particles will adhere together. The vapor is introduced into the gas to provide a gas-to-vapor ratio which, within the range of temperatures employed, will result in surface condensation and/or absorption of the vapor upon the particles in the upper strata of the bed of moving particles.

In this Specification and appended claims the "vapor-gas" consists either of condensable vapors alone in superheated condition or of said superheated and condensable vapors admixed with a substantially non-condensable carrier gas.

Where herein and in the appended claims the term "fluidization" is used, the broad significance of that term is intended to include not only the condition which is known in the process industries as "fluidization" of pulverulent solids, but also a condition where the solid particles are partially suspended in the gaseous mixture by mechanical agitation or other means to an extent necessary to cause them to flow in much the same manner as a liquid.

The basic principle of our process in the preferred form is described and illustrated in more detail by reference to Figure 4 of the drawings. It will be noted that the uniformly introduced, pulverulent material is fluidized into the form of a bed which is caused to travel through a predetermined, generally horizontal path in the apparatus illustrated.

It will of course be understood that variations in said general path such as declinations or breaks in the continuity thereof from screen to screen may be employed without departing from the scope of our invention.

The superheated vapor-gas supplied from the lower chamber 31 flows upwardly through the permeable supporting medium or sheet 18 and through a particular area of the moving bed thereabove, and is initially maintained at a temperature considerably above the dew point of the vapor-gas utilized as will later be indicated from the ranges of temperature expressed in the examples accompanying this Specification. In said upward flow of vapor-gas, the superheat is dissipated by the underlying layer of the bed of fluidized particles and heats the same until, after dissipation of the superheat, condensation occurs in the upper portion of the moving bed and preferably in a stratum of a thickness of several times the thickness of the dry underlayer, as indicated in the diagrammatic illustration, Figure 4. The condensate on the surfaces of the particles causes said surfaces to become softened and adherent so that agglomeration proceeds in the said upper stratum with the rapid agitation and dispersion of particles effected through continued fluidization of the moving bed of material. Such fluidization is preferably brought about through the upward flow of gas or gaseous medium in the treating chambers and, in certain applications of our process, in the preliminary fluidization chamber 29.

It is essential that the temperature of superheat of the vapor is sufficiently above the dew point of the vapor-gas to prevent condensation and/or absorption of the agglomerating vapor close to the porous medium or sheet.

The dispersion and agitation of the solid particles (powdered and initial agglomerates) and the efficiency of agglomeration carried out in the first treatment chamber 33*a* and in the sectond or drying treatment chamber 38 are substantially enhanced by the rapid, short-stroke vibration of the apparatus preferably including not only vibration of the permeable supporting medium or sheets but also of the chamber-defining walls of the apparatus, in directions generally transverse to the upwardly flowing vapor-gas. If in chamber 33*a* certain of the discrete pulverulent particles or adhered pluralities of such particles in the upper stratum carrying adhesive surface films shift downwardly in the resulting dispersion, the same encounter dry heated particles disposed therebelow and pick up numbers thereof in their subsequent general horizontal travel.

By reference again to Figure 4 it will be seen that the fluidized bed moves generally horizontally from the first treatment chamber 33*a* into the drying treatment chamber 38 where a drying gas usually but not necessarily maintained above room temperature is flowed upwardly through the solid material which is then for the most part in the form of agglomerates. The inclined line L indicates approximately the plane of the moving bed in the drying chamber 38 above screen 18 beyond which almost all of the agglomerates are rigidified and the adhesive substances hardened.

In many applications of our process it is desirable to cool the dry agglomerated material which discharges with the moving bed from the drying chamber 38 and thus, in the apparatus of Figures 1 to 3, a cooling chamber 43 is employed above a permeable supporting medium or sheet 20 through which dry, inert gas such as air is flowed upwardly at the desired cooling temperature.

The use of the coarser classifying sheet 21 and the discharge spouts 46 and 48 is optional but is desirable for production of agglomerates

melting of the surfaces or by premature condensation of the agglomerating vapors thereon. In general, therefore, the gas used can be at any temperature below the melting point or softening point of the material being treated, and above the actual temperature of the pulverulent material at the time of treatment.

Some organic materials do not become sticky when moistened with a liquid such as water. Lard flakes, as an example, do not become adherent through the surface condensation of water vapor, but many organic solvents such as hydrocarbons do possess the necessary solvent action and can be used as the agglomerating vapor. The operating conditions must be so chosen, as heretofore described, to achieve surface condensation in the agglomerating zone so that surface softening, adhesion, and agglomeration can proceed.

EXAMPLE 2—CHOCOLATE-DRINK-PRODUCT.

A powdered chocolate drink type product consisting of 20% cocoa powder and 80% finely ground sugar was agglomerated using 4.0 pounds of steam per minute mixed with 8.5 pounds per minute of air. The gas-vapor mixture was heated to 225° F. prior to entering the treating chamber. A frequency of vibration of 1050 cycles per minute and a vibration amplitude of $^{3}/_{16}$ inch were used. The dry powder was supplied to the agglomerator at a rate of 8.5 pounds per minute.

Sieve analysis.	Agglomerated.	Non-agglomerated.
U.S. Sieve No.		
over 14	11%	0%
14—36	41%	0%
36—60	24%	0%
60—100	16%	0%
Finer than 100	8%	100%
Bulk density (tapped to constant volume)	0.43 g/cc.	0.74 g/cc.

To test the ease of dispersion of the agglomerated and non-agglomerated powders in cold milk, a teaspoon of each was placed on the surface of cold non-agitated milk. The time required for the powder to become submerged below the surface was noted and was 2 seconds for the agglomerated chocolate drink powder and in excess of 5 minutes for the non-agglomerated powder. Upon stirring, the non-agglomerated product formed nearly insoluble pills which could not be readily dispersed. The agglomerated product, on the other hand, with only slight stirring, dispersed into a uniform suspension in which very little settling out or floating of the particles occurred during subsequent standing. As an instant type of beverage preparation this suspension possessed all the usually desirable characteristics, whereas the mixture of non-agglomerated product with milk neither looked nor tasted like a chocolate beverage because of the failure of the solids to disperse adequately.

EXAMPLE 3—FLOUR-SUGAR MIXTURES.

Mixtures of flour and powdered sugar were agglomerated. The effect of sugar-flour ratio and agglomeration on the bulk density and dispersion time in water is shown in the table below. The amount of steam used was adjusted for each run so as to effect good agglomeration in each case. During the several runs hereafter tabulated the temperature of the vapor-gas mixture passing upwardly through the screen of the agglomerating chamber was approximately 230° F.

Sugar %	Flour %	Pounds of steam per pound of dry air.	Dew point ° F.	Bulk density—g/cc. Not Aggl.	Bulk density—g/cc. Agglom.	Dispersion time (sec.). Not agglm.	Dispersion time (sec.). Agglm.
90	10	0.65	180	0.661	0.433	22	1
75	25	0.72	182	0.726	0.466	22	1
50	50	0.83	185	0.705	0.398	22	1
25	75	0.79	184	0.699	0.387	26	2
15	85	0.90	186	0.681	0.447	28	7
10	90	1.03*	189	0.685	0.541	30	10
5	95	1.03*	189	0.669	0.553	35	20

* Maximum obtainable in apparatus.

of specified size with many types of pulverulent material.

EXAMPLES.

To illustrate the manner in which various materials can be agglomerated with apparatus of the type we have invented and herein described, to demonstrate the utility of and means for controlling the process variables, and to point out the benefits attained when powdered materials are treated in accordance with our process, there follows a series of examples of agglomerating tests made with a pilot plant apparatus constructed and operated as heretofore described, the essential dimensions and features of apparatus common to all examples being :—

Width of agglomerating section .. $5\frac{1}{4}$ inches
Length of agglomerating section .. $9\frac{1}{2}$ inches
Vibration—30 degrees from horizontal in direction of product travel.

EXAMPLE #$\frac{1}{4}$—ANGEL FOOD CAKE "A" MIX.

In this example, the material agglomerated was a finely powdered mixture, an angel food cake "A" mix, consisting of :—

Dried egg albumen	31.258%
Powdered sugar	67.308
pH control agent, anhydrous monocalcium phosphate ..	1.434
	100.000%

Other conditions were as follows:—

	Pounds per minute.
Feed rate of powdered material	10
Carrier gas, air	9.2
Stroke, $\frac{1}{4}$ inch vibration of frame cycles per minute	800
	Pounds per minute.
Agglomerating vapor, steam ..	3.5
	° F.
a	189
b	202
c	212
d	223
e	228
f	240
Dew point of gas mixture fed	166

In this test heavy fouling of the pervious sheet occurred at a gas temperature of 189° F., and the extent of fouling decreased at the successively higher temperature until, at 223°, the fouling disappeared.

At all higher temperatures the sheet remained completely clean, and agglomeration proceeded in a highly efficient manner, as evidenced by the granular nature of the agglomerated product when compared to the dusty nature of the fine powder before agglomerating and by greatly improved water dispersibility of the agglomerated material.

The reason why the sheet fouled at the lower gas temperatures, as demonstrated by these data, is that the temperture was not sufficiently above the dew point. The small amount of superheat in the agglomerating vapor was dissipated so rapidly upon contact with the cooler powder in the lower layer that the gas temperature dropped rapidly to the dew point. Condensation and absorption of the condensed liquid then occurred so that the particle surfaces became sticky in the lower layer, and the sheet became fouled.

At the higher temperatures, the amount of superheat in the gas was sufficient to prevent condensation until the gas reached a higher layer. The lower layer material of course cooled the gas to some extent, but the dew point, or temperature at which appreciable condensation occurred, existed only in the upper layers. The lower layer remained dry and in a substantially fluidized state to provide the underlying bed of flowable material, on top of which the particles being moistened and rendered sticky would be supported for movement toward the discharge end.

As the material flowed toward the discharge end of the apparatus, the sticky upper and intermediate layers comingled increasingly with the dry lower layer so that substantially all the particles had opportunities to collide in such a manner as to adhere together in the form of discrete agglomerates.

COMMENT.

Numerous tests have been made to demonstrate that the presence of the superheated agglomerating vapor is necessary to accomplish the agglomeration. When it is not present, as in the case when only hot air is used, the powdered material simply travels across the sheet without forming agglomerates. When the agglomerating vapor is employed with insufficient superheat, fouling of the equipment occurs, as in the initial parts of the preceding example.

If a thermoplastic material is treated according to the preferred form of our process, the gas temperature must not be so high as to melt the material. This has been demonstrated repeatedly, for example, with pulverized hydrogenated lard flakes and glyceryl monostearate. A gas temperature high enough to soften the surface by heat alone causes an immediate breakdown of the lower fluidized layer so that the equipment fouls and becomes inoperative. It is essential to avoid any conditions that cause stickiness in the lower layer, such as by incipient

EXAMPLE 4—POWDERED SUGAR ALONE.

Powdered sugar was agglomerated using three different steam/air ratios and two different vibration frequencies. The effect on particle size distribution of the agglomerated products is shown below. During these runs the humid air temperature was held at 230° F. and the air flow at 8.5 pounds/minute. A $\frac{3}{8}$ inch amplitude of vibration was employed and the sugar was supplied at a rate of 12 pounds per minute to the $5\frac{1}{4}$ inches wide and $19\frac{1}{2}$ inches long agglomerating section.

	Run I.	Run II.	Run III.
Steam—pounds per minute	2.9	2.9	3.13
Steam/air ratio	0.34	0.34	0.37
Frequency of vibration cycles per minute	900	800	800
Particle Size—U.S. Sieve No.			
Larger than 12	21.6%	14.6%	27.6%
12—16	21.4	22.0	23.3
16—20	23.5	16.2	15.4
20—40	18.9	20.5	20.5
40—60	8.0	9.1	5.3
Finer than 60	6.6	17.6	7.9

All of the sugar passed through a #60 sieve before agglomeration.

The agglomerated sugar from the three runs dispersed and dissolved instantly in water whereas the unagglomerated powder required over 60 seconds to become dispersed and dissolved.

The above example clearly shows two separate means whereby the particle size distribution of the agglomerates may be adjusted, namely by means of the steam rate and by means of the frequency of vibration. The finer overall particle size in Run II as compared to Run I was accomplished by using a lower frequency of vibration. By increasing the steam rate, as in Run III, a size range is again obtained which approximates the results of Run I quite closely although the vibration rate was the same as in Run II.

EXAMPLE 5.

Crystalline Ammonium Sulfate was ground on a "Mikropulverizer" (Registered Trade Mark) using a screen with 0.039 inch round perforations. The agglomerator was operated at 1000 cycles per minute, $\frac{1}{2}$ inch amplitude, 10 pounds per minute feed rate. A mixture of 4 pounds steam and 8.5 pounds air per minute heated to 220° F. was supplied to the agglomerating section. The bulk density of the material was lowered substantially, as follows:—

	Grams/cc.
Ammonium Sulfate—Crystalline	1.06
Ammonium Sulfate—Ground	0.89
Ammonium Sulfate—Ground—Agglomerated	0.59

The agglomerated powder dispersed and dissolved instantly in cold water whereas the crystalline material dissolved slowly because of the large crystal size, and the ground material dispersed and dissolved very slowly because of poor wettability.

EXAMPLE 6.

A mixture containing 32% dried egg albumen and 68% powdered sugar was agglomerated using three different steam-to-air ratios, all other factors being held constant. The table below shows the reduction in bulk density and dispersion time in each case. In each instance the temperature of the vapor-gas mixture was supplied and maintained at 230° F.

Steam/air ratio.	Bulk density grams/cc.	Dispersion time seconds.
0.34	0.404	40
0.40	0.359	25
0.47	0.318	20
Non-agglomerated	0.664	Over 60*

*Dispersion times in excess of 60 seconds not measured.

After five months storage in a cardboard package with glassine liner under 100° F. and 50% relative humidity, the non-agglomerated product was badly caked. All agglomerated products remained free flowing under the same conditions.

EXAMPLE 7.

Sodium Hexametaphosphate was ground through a screen with 0.020 inch round perforations. The powdered material was agglomerated using 1.75 pounds/minute steam mixed with 8.5 pounds/minute air. This mixture was heated to 225° F. The feed rate of the Hexametaphosphate was 10.5 pounds per minute. The agglomerator operated at a frequency of 1050 cycles per minute and $\frac{1}{4}$ inch amplitude of vibration.

Sieve analysis :—

U.S. Sieve No.	Agglomerated. %	Non-agglomerated. %
over 14	30	0
14—36	29	0
36—60	10	0
60—100	17	0
Finer than 100	14	100
Bulk density (tapped to constant volume)	0.60 g/cc.	1.06 g/cc.

A simple dispersion test was conducted on both the agglomerated and non-agglomerated powder as follows :—

One teaspoon of the powder was dropped into one glass of cold water. The time for complete dissolution of the powder was noted. No agitation was used.

In the case of the non-agglomerated powder, the material sank to the bottom of the glass where it stayed substantially undissolved for a period in excess of 5 minutes at which time the test was discontinued. The agglomerated product appeared completely dissolved after a period not exceeding 5 seconds. Initial cloudiness created by the agglomerated powder in the water prevented a more exact time determination, but at the end of said interval of 5 seconds the water was crystal clear, all of the powder having gone rapidly into solution.

WHAT WE CLAIM IS :—

1. A process of agglomerating pulverulent material including the steps of continuously dispersing said material so as to fluidize the particles of said material and form a fluidized bed of said material in which the particles are dispersed and suspended in intimate relationship with one another and make repeated contact with one another, maintaining said material within the confines of said bed while moving the bed over a supporting medium, and subjecting the fluidized particles of at least a part of said moving bed to a condition changing agent so as to form adhesive agglomerating surfaces on the particles and thereby effect agglomeration thereof.

2. A process according to Claim 1, wherein the supporting medium is permeable, said process including the step of passing a gaseous medium through said bed and said permeable supporting medium to form the adhesive agglomerating surfaces on the particles.

3. A process according to Claim 2, including the step of passing a superheated vapor gas through said bed and said permeable supporting medium and thereby causing condensation of said vapor gas on the surfaces of the particles to produce the adhesive agglomerating surfaces thereon.

4. A process according to any of Claims 1 to 3, including the step of agitating said bed during said movement without substantially deforming the cross sectional areas of said bed.

5. A process according to Claim 2 or 3, wherein the bed is rapidly and continuously vibrated along directions generally transverse to the flow of said gaseous medium or vapor gas.

6. A process of agglomerating pulverulent materials, including the steps of continuously dispersing said materials to produce a fluidized bed of such materials on a supporting medium, moving said bed substantially continuously along a predetermined course of travel on said supporting medium, maintaining the particles in at least the upper stratum of said bed in an agitated and dispersed state, forming adhesive surfaces on said agitated particles and thereby causing agglomeration thereof, and simultaneously maintaining the particles in the lower stratum of said bed next adjacent said supporting medium in a substantially non-adhesive

condition during the agglomeration of said upper stratum.

7. A process according to Claim 6, including the step of flowing a superheated vapor-gas through said bed at a temperature originally above the dew point of said vapor-gas for heating the lower stratum of said bed and in so doing reducing the vapor-gas temperature to at least its dew point and thereby depositing condensation on surfaces of the particles in the upper stratum of the bed for forming adhesive agglomerating surfaces thereon.

8. A process according to Claim 7, wherein the supporting medium comprises at least two adjacent sections, the first of said sections being highly permeable, said process including the step of moving said bed of pulverulent material successively over said supporting sections, flowing said superheated vapor-gas through said first permeable supporting section to partially suspend and disperse the particles in said moving bed and condensing vapor on the surface of particles in the upper stratum of said bed thereby producing adhesive films on the surfaces of particles, and flowing a drying medium through the bed of material moving above said second supporting section, to dry said adhesive film and rigidify the agglomerates formed.

9. An agglomerated product as made in accordance with the process claimed in any of the preceding claims.

10. A product according to Claim 9, wherein the pulverulent particles comprise sugar particles.

11. The product according to Claim 9, wherein the pulverulent particles comprise sodium hexametaphosphate particles.

12. The product according to Claim 9, wherein the pulverulent particles comprise ammonium sulfate particles.

13. A product according to Claim 9, comprising a mixture of pulverulent material including dried egg albumen and sucrose which together constitute the major portion of said mixture, the particles comprising said mixture being bonded to each other at their interfaces by an adhesive film formed from said particles and forming porous lacy agglomerates, said agglomerates being free flowing non-caking and quickly dispersible in a liquid.

14. The product of Claim 13, in which the agglomerated mixture includes a *p*H control agent.

15. The product of Claim 14, in which the control agent is anhydrous monocalcium phosphate.

16. The product of Claim 13, in which the agglomerated mixture contains a higher percentage of sucrose than dried egg albumen.

17. The product of Claim 13, wherein the ratio of sugar to dried egg albumen is approximately 2 to 1.

18. The product of Claim 13, wherein the mixture is composed of approximately 32% dried egg albumen and 68% sucrose.

19. A product according to Claim 9, comprising a mixture of pulverulent particles in the form of porous lacy agglomerates, in which the particles are bonded to each other at their interfaces by an adhesive film formed from said particles, said mixture being composed of pulverulent cocoa and sugar particles, with the sugar constituting the major portion thereof, said agglomerates being free flowing, non-caking, and quickly dispersible in a cold liquid.

20. The product of Claim 19, wherein the mixture comprises 20% cocoa and 80% sugar.

21. Apparatus for carrying out the process claimed in any of the preceding Claims 2, 3, 7 or 8, including means for flowing the material-dispersing gaseous medium or vapor gas upwardly through a first portion of the area of the permeable supporting medium adjacent a receiving end thereof, said supporting medium forming the floor of an elongated, channel-shaped structure through which the pulverulent material flows, the upper part of said structure being closed by a top member, the undersurface of said top member having at least one baffle extending downwardly therefrom to a point spaced from the structure floor and said baffle comprising the only obstruction to the movement of material over the floor.

22. Apparatus according to Claim 21, wherein said structure is provided with one or more supports for pivotally connecting said structure to a fixed base and motor means drivingly coupled to said structure for imparting rapid vibratory movement thereto.

23. Apparatus according to Claim 21 or 22, wherein said structure includes a bottom member secured along its side edges to the side walls of said structure in spaced relation to said supporrting medium, said bottom member having at least one upstanding baffle extending between said member and the supporting medium to define one or more sealed chambers in the space therebetween, and conduit means for connecting a source of superheated steam to the chamber beneath the first portion of said supporting medium.

24. The process for agglomerating pulverulent materials substantially as herein described.

25. An agglomerated product substantially as herein disclosed with particular reference to the accompanying examples in the disclosure.

26. Apparatus for agglomerating pulverulent material constructed substantially as herein described with reference to the accompanying drawings.

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Agents for the Applicants.

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FIG. 1

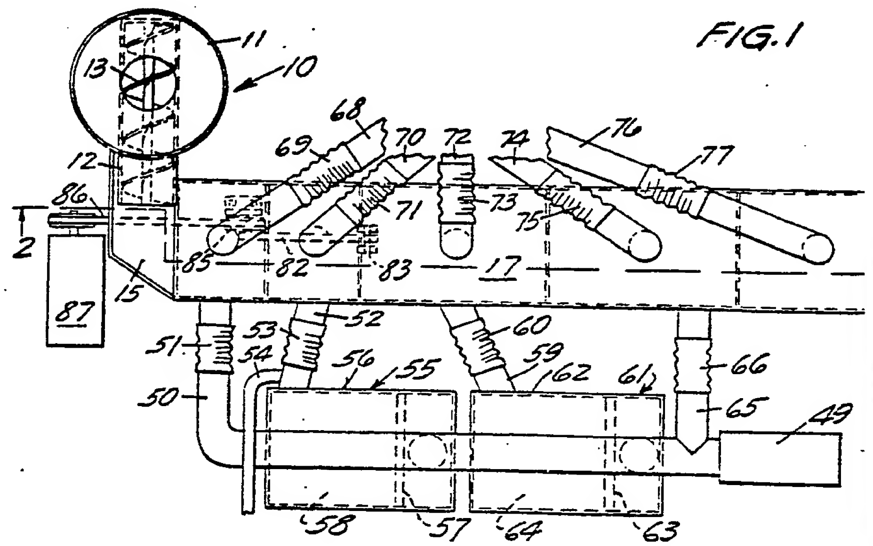
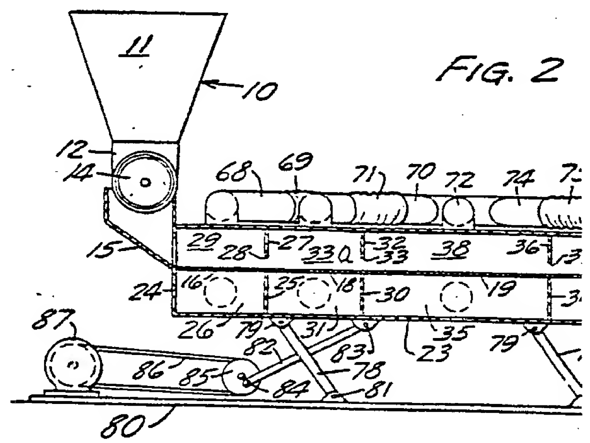


FIG. 2



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Sheet 1

FIG. 1

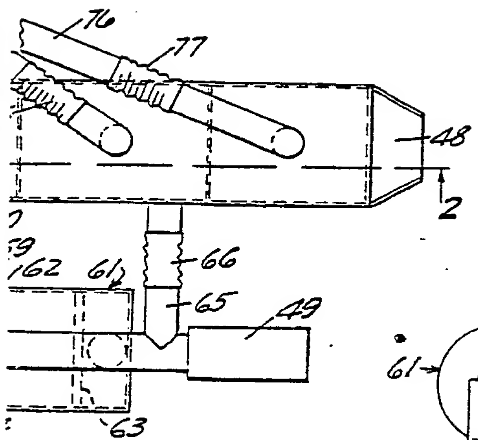


FIG. 3

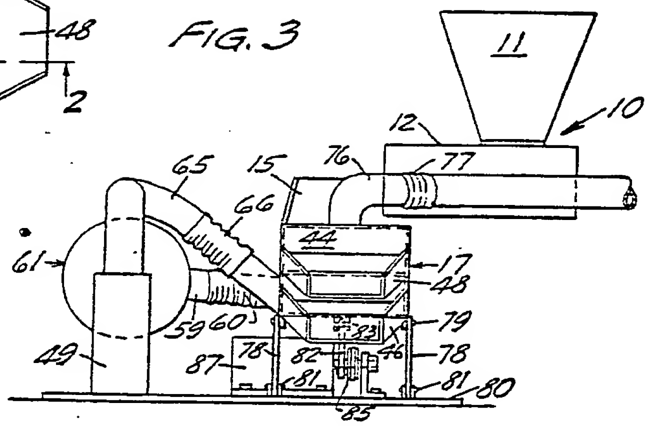
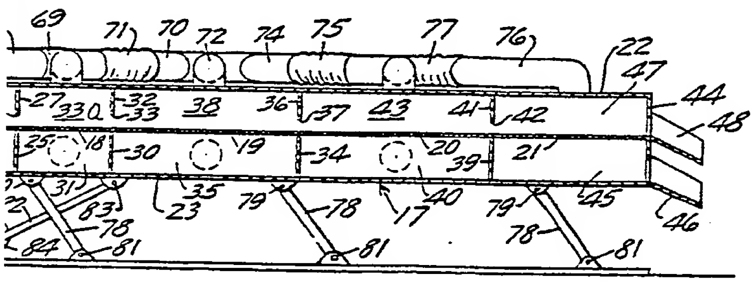


FIG. 2



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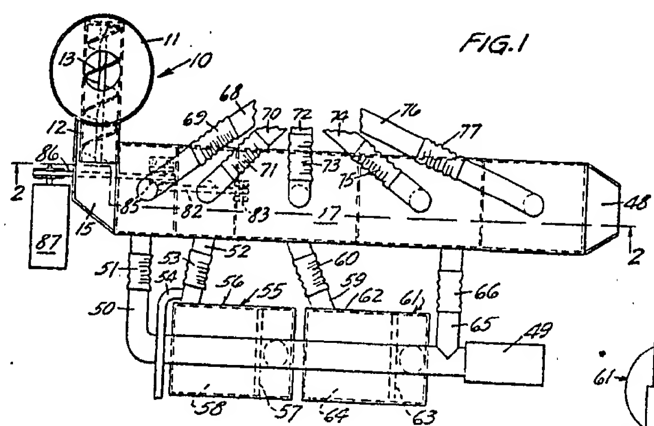
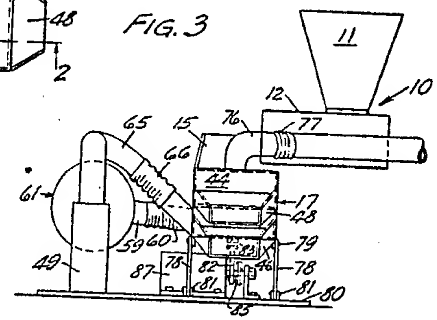


FIG. 3



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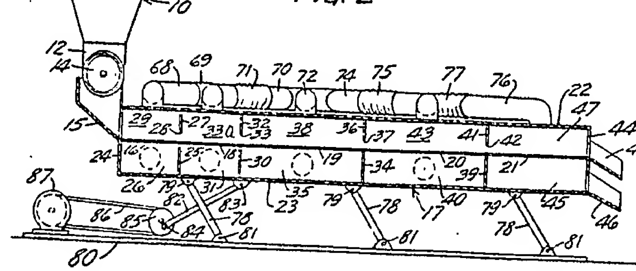
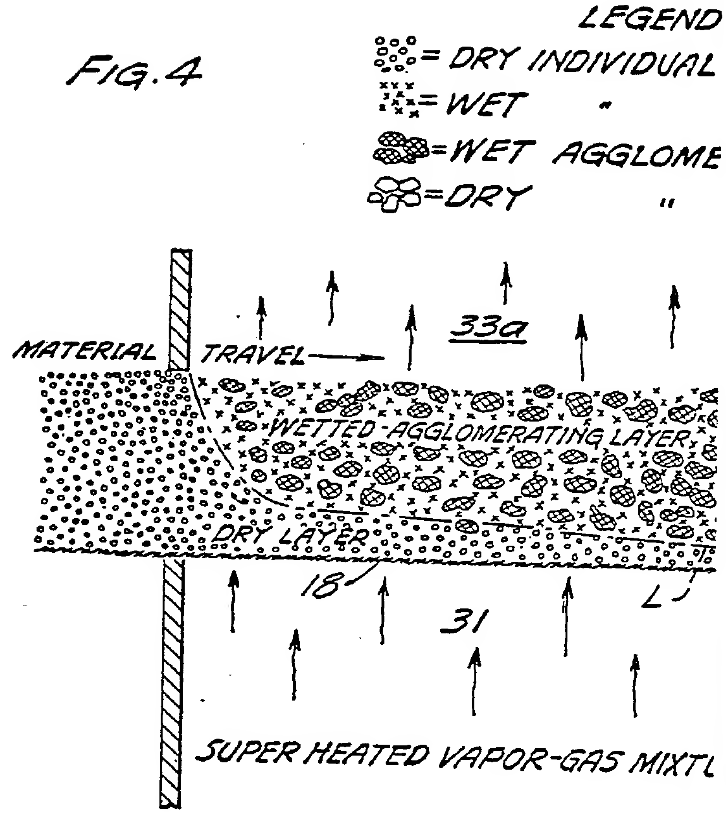


FIG. 4


LEGEND
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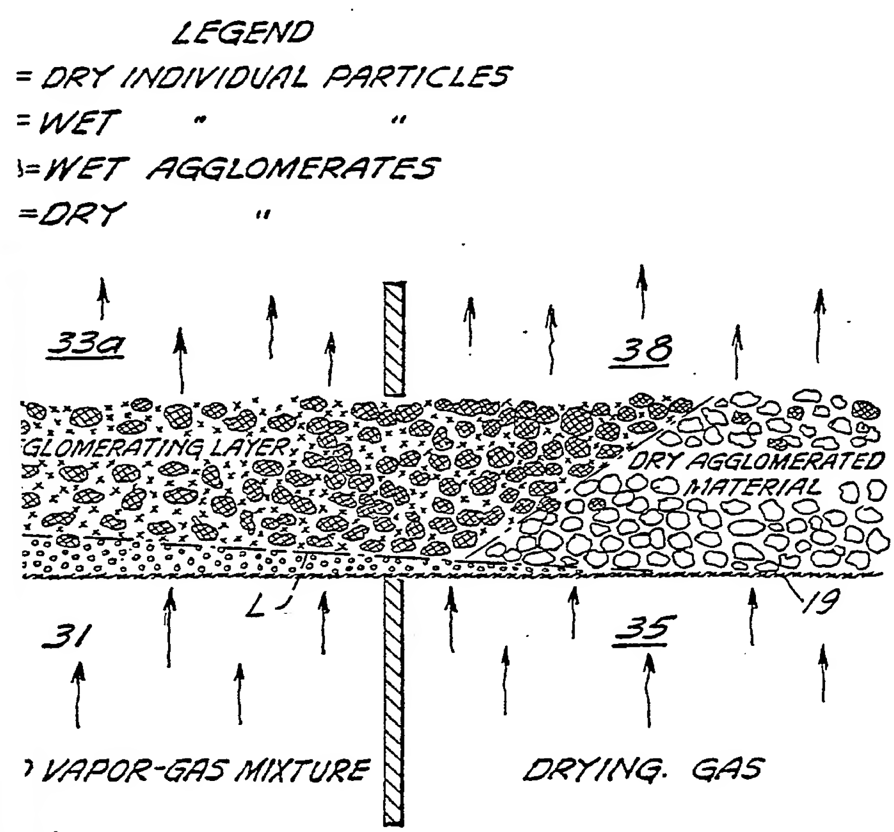
Sheet 2



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Sheet 2

FIG. 4